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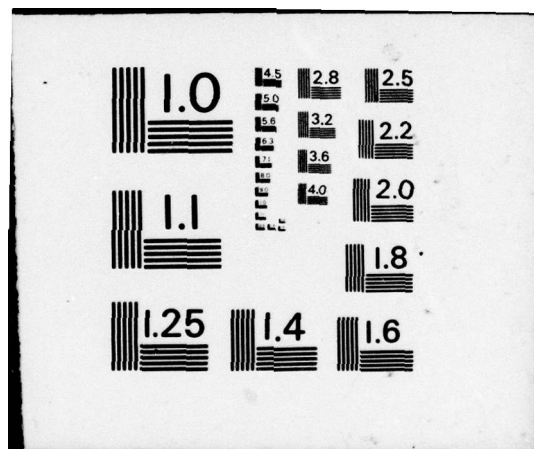   DEC 76 F L RODKEY, A R DASLER

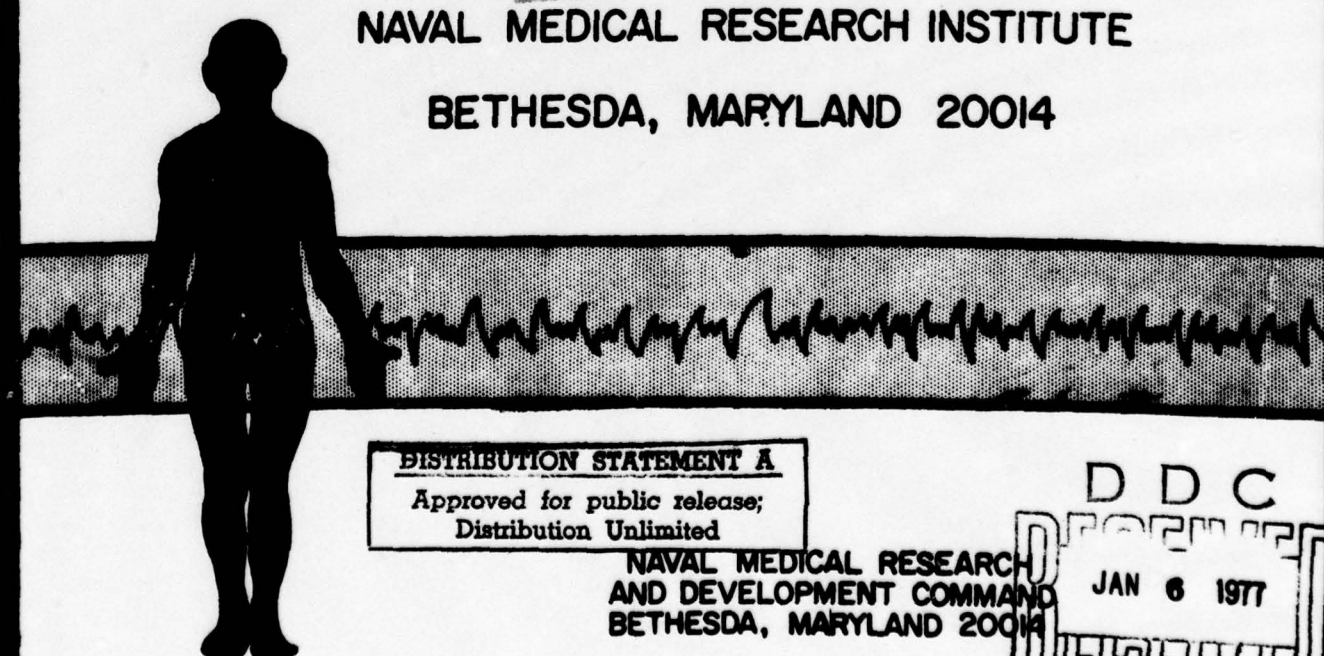UNCLASSIFIED   NL

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A METHOD FOR ANALYSIS OF OXYGEN BREATHING APPARATUS (OBA)

TYPE II CANISTERS

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and K_2CO_3, formed upon dissolving the canister contents, permitted quantitation of the amount of CO_2 absorbed during canister use and the total amount of O_2 available in the unused canisters. The charge composition of the Type II canisters, on a mole fraction basis, was estimated by analysis of 17 unused canisters and was found to be 92% KO_2, 7% K_2O_2 and 1% K_2CO_3. The method was used to determine the amount of O_2 released and the amount of CO_2 absorbed during manned use of the OBA Type II canisters.

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December 1976

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ABSTRACT

A METHOD FOR ANALYSIS OF OXYGEN
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A method was devised to rapidly release the chemical reaction products from Oxygen Breathing Apparatus (OBA) Type II canisters in a closed system. This procedure permitted accurate chemical analyses without the atmospheric interferences inherent in the conventional approach to OBA canister analyses. The available O_2 was released by immersion of the entire canister, with its charge intact, in a known amount of water, in such a manner that all O_2 was collected and measured volumetrically. Analysis of the water for total KOH and K_2CO_3, formed upon dissolving the canister contents, permitted quantitation of the amount of CO_2 absorbed during canister use and the total amount of O_2 available in the unused canisters. The charge composition of the Type II canisters, on a mole fraction basis, was estimated by analysis of 17 unused canisters and was found to be 92% KO_2, 7% K_2O_2 and 1% K_2CO_3. The method was used to determine the amount of O_2 released and the amount of CO_2 absorbed during manned use of the OBA Type II canisters.

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INTRODUCTION

The OBA is a self-contained, closed-circuit device for life support during physical activity ranging from rest to heavy work. Oxygen is generated from chemicals within a canister and exhaled carbon dioxide is absorbed. The "Quick-Start" Type II canister is widely available today. This type of canister contains a percussion cap activated chlorate candle to provide an initial 10 liter "burst" of oxygen. The major portion of the chemical charge within a canister is composed of potassium superoxide (KO_2) which reacts with water from the operator's exhaled breath to liberate oxygen according to Equation 1.[1]

$$4 \; KO_2 + 2 \; H_2O \rightarrow 4 \; KOH + 3 \; O_2 \qquad (1)$$
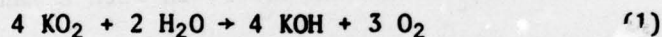
Exhaled carbon dioxide reacts with KOH formed in this reaction to yield potassium carbonate according to Equation 2.

$$2 \; KOH + CO_2 \rightarrow K_2CO_3 + H_2O \qquad (2)$$
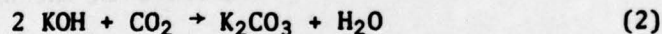
Water produced in reaction 2 is utilized in the liberation of additional oxygen.

It is sometimes necessary to analyze unused and used OBA canisters in order to estimate three significant parameters: 1) the original available oxygen in unused canisters, 2) the residual available oxygen in used canisters and 3) the total carbon dioxide

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absorbed during use of canisters. This report presents a rapid, convenient method for release and analysis of the contents of OBA canisters. Oxygen produced by the "Quick-Start" chlorate candle is not included in the measurements herein.

APPARATUS

The analysis of residual available oxygen (defined as the oxygen which can be liberated by water) is done by completely filling the canister with water and collecting all of the gas released. As the gases are released the volume may be measured by collection in a large spirometer or by passage through a gas flow meter; however, for analytical purposes collection of the gases in a spirometer is highly recommended. A tank containing a known volume of water (30 liters) was used in conjuction with a 5 gallon reagent container (hereafter called a "bell") to liberate and collect all of the released oxygen, (Figure 1). The bottom of the bell was removed and a support provided to hold the canisters in an upright position near the inside tope of the bell. The pouring spout of the bell was connected directly to the spirometer, or to a flowmeter. Use of a spirometer was preferred as this approach of measuring gas volume was unaffected by the rate of oxygen release which varied considerably during the reaction of the chemicals and water; furthermore, the rate of release of gases may exceed the capacity of the flowmeter.

PROCEDURE

Weight of Canister Charge. Weigh the canister before analysis.

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After analysis the canister must be dried at 110°C for 6 - 12 hours and again weighed. The difference in weight is the weight of the canister charge.

Canister Available Oxygen. Remove the neck seal and punch 6 - 8 large holes (as with a triangular beer can opener) in the bottom of the canister. Punch 6 - 8 holes on the side of the canister, using a screwdriver or a cold chisel. The holes on the side of the canister must be within the confines of the charge bed. These holes should be in two rows about 4 cm and about 11 cm from the top of the canister body. Reaction with water is too slow and incomplete unless an adequate number of large holes are made in the canister shell. Fasten the canister in an upright position inside the gas collecting bell and connect the outlet of the bell to the spirometer. Place the bottom of the bell just under the surface of the water (30 liter known volume) in the tank. Tap water was shown to be satisfactory for use in this study. Immediately take the following readings: 1) initial spirometer value (mm), 2) barometric pressure (mm Hg) and 3) spirometer temperature (°C).

Depress the bell into the water until the canister is partially submerged and oxygen liberation starts. Gradually lower the bell into the water until the canister is completely submerged and all oxygen is liberated. Gentle shaking of the bell will sometimes aid in dislodging pockets of chemical charge which may be unreacted. When all oxygen has been released and the bell is filled with water, make the following

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readings: 1) final spirometer value (mm), 2) barometric pressure
(mm Hg) and 3) spirometer temperature (°C). The actual volume of
the bell (V_b) must be known from previous measurements; a bell of
approximately 19 liters volume is required.

The available oxygen, saturated with water at ambient temperature
and pressure (ATPS), is calculated from Equation 3;

$$O_2 \text{ (Liters ATPS)} = (R_f - R_i) F_s - V_b \qquad (3)$$

where, R_i and R_f are the initial and final spirometer reading respect-
ively, F_s is the spirometer factor in liters/mm, and V_b is the volume
of the bell used to hold the canister. The available oxygen is con-
verted to standard temperature and pressure (STPD) by Equation 4;

$$O_2 \text{ (Liters STPD)} = O_2 \text{ (Liters ATPS)} \times \frac{P_b - P_{H2O}}{760} \times \frac{273}{273 + t} \qquad (4)$$

where, P_b and t are the measured ambient barometric pressure (mm Hg)
and temperature (°C), and P_{H2O} is the vapor pressure of water at the
ambient temperature. Available oxygen may be expressed in molar
quantities since 22.4 liters STPD is equivalent to 1.0 mole.

<u>Canister Carbon Dioxide Content</u>. After all oxygen has been
released and the gas volume measurements taken, the 30 liters of water
must be carefully mixed by raising and lowering the bell to force the
water to rinse in and out of the canister. A uniform solution must
be obtained containing all of the water soluble components from within
the canister. Finally, raise the bell above the water to drain and

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remove the canister. Stir the solution thoroughly (CAUTION: THIS IS
NOW A HIGHLY CAUSTIC SOLUTION OF KOH AND K_2CO_3) and save a portion of
about 250 ml in a polyethylene bottle for analysis. The remaining
solution must be carefully discarded and the tank and bell washed in
preparation for another analysis. Rinse the canister under tap water
and place it in a drying oven at 110°C to dry before obtaining the
final weight of the canister.

Two analyses are performed on the 250 ml aliquot of the caustic
solution. One portion (A) of the solution (15 ml) is titrated with
standard 1.0 N HCl to pH = 8 using phenolphthalien as the indicator.
The normality of the solution (N_a) is calculated. This represents
titration of all free KOH and the titration of K_2CO_3 to $KHCO_3^-$.
Hence, the total titration;

$$N_a \times V_s = M_{KOH} + M_{K_2CO_3} \qquad (5)$$

where V_s is the volume of the solution in liters and the total amounts
of KOH and K_2CO_3 in the solution are expressed in moles. The second
portion (B) of the caustic solution (15 ml) is treated with 0.5 - 0.75
gm of solid $BaCl_2$ and mixed to precipitate the carbonate as barium
carbonate. This mixture is then titrated with standard 1.0 N HCl to
pH = 8, using phenolphthalien as the indicator. The normality of the
solution (N_b) is calculated. As the $BaCO_3$ is not titrated under
these conditions, the titration represents free KOH only, hence;

$$M_{KOH} = N_b \times V_s \qquad (6)$$

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The total moles of CO_2 in the canister is therefore,

$$M_{CO_2} = (N_a - N_b) V_s \qquad (7)$$

The CO_2 may be expressed in liters since 1 mole of CO_2 is equivalent
to 22.26 liters STPD.

RESULTS

Original Available Oxygen. Unused canisters contain only KO_2
with minor impurities of K_2O_2, KOH and K_2CO_3. All of these components
will dissolve in water to give KOH or K_2CO_3 in the final solution. Only
KO_2 and K_2O_2 will contribute to the available oxygen. The titration
values of the water solution can be used to express the total number
of moles of base which is assumed to be derived from three sources;
1) a small amount of K_2CO_3 in the original canister charge, 2) hydration
of KO_2 (and K_2O_2) during both use of the canister in the OBA and during
the analytical total hydration, and 3) the K_2CO_3 formed by absorption
of carbon dioxide during use. With these assumptions the total
apparent or equivalent moles of KO_2 in the original canister is the sum
of the free base titrated, M_{KOH}, plus the hydroxide which has reacted
with the CO_2 absorbed, i.e. $2 (M_{CO_2} - M_{CO_2 \text{ blank}})$ as given in Equation
8;

$$M_{KO_2} = M_{KOH} + 2 (M_{CO_2} - M_{CO_2 \text{ blank}}) \qquad (8)$$

The apparent purity, P, of the original KO_2 canister charge, based on
available oxygen, M_{O_2}, and the $M_{CO_2 \text{ blank}}$ were determined by analysis
of a series of 17 new, unused canisters, Table 1. The relation

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$M_{O_2} = (0.75 \text{ X } M_{KO_2} \text{ X } P)$ was used and M_{CO_2} blank was the observed M_{CO_2} in unused canisters. Actual oxygen, M_{O_2}, was liberated and measured in the spirometer. The mean value of M_{CO_2} blank was 0.12 (±0.014 SEM) with a range of 0.05 to 0.25. The mean value of P was 0.908 (±0.003 SEM) with a range of 0.879 to 0.928. Thus, the apparent purity of the canister charge as KO_2 was 91%. Hence, the original available oxygen (in new and used canisters) was calculated from Equation 9;

$$M_{O_2} = [M_{KOH} + 2 (M_{CO_2} - 0.12)] \, 0.75 \text{ X } 0.91 \qquad (9)$$

where 0.75 is the moles of O_2 released per mole of KO_2 and 0.91 is the apparent purity of KO_2 in new canisters.

It should be noted that the "apparent purity" will reflect the presence of other alkali metals, such as sodium, or other oxides such as K_2O_2, K_2O, or KOH. Data from the 17 unused canisters including charge weight, available oxygen, and the KOH plus K_2CO_3 obtained by solution were consistent with an initial charge composition on a mole fraction basis of 92 ± 0.4% KO_2, 7 ± 0.3% K_2O_2, and 0.9 ± 0.1% K_2CO_3. This composition of the canister charge leads to a calculated apparent purity of 91.2% as KO_2, based upon O_2 released, as compared with the mean value of 90.8% measured from the new canisters.

Oxygen Liberated from the Chlorate Candle. The oxygen produced by the buring chlorate candle is not measured in the present method of hydration of the canister charge. A separate group of eight unused

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canisters was tested to determine the volume of oxygen released by the chlorate candles. Four of the canisters were in an upright position during activation and operation of candle combustion and four canisters were placed on their broadest side for similiar operations. Position differences did not alter the amount of oxygen released nor the physical appearance of the candles after burning. A mean of 10.2 ($\pm$0.1 SEM) liters STPD oxygen release was observed for the eight candles. The candles were activated when the canisters were maintained at room temperature (25°C) and oxygen release was complete in every case within 1.5 minutes after the percussion cap was fired.

Analysis of Used Canisters. A series of canisters were analyzed after varying lengths of manned-use time in OBA's. Residual oxygen was measured by collection in the spirometer when the contents of the used canisters were completely reacted with water. Free KOH and total K_2CO_3 were used to estimate the original amount of oxygen available and the amount of carbon dioxide absorbed at the time of terminating use of the canisters. Subjects using OBA's were exercised at a time-weighted-mean O_2 consumption of 1.5 liters/minute STPD in accordance with the procedures described by Dasler.[2] Results from analyses of the manned-use canisters are given in Table 2. When the canisters operated with minimal physiological strain in the subjects the chemical analyses revealed that as much as 72% of the original oxygen charge was liberated from the canisters. There were two canisters which precipitated extreme physiological strain at about

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20 minutes of what should have been at least 60 minutes of manned-use.
The two specific canisters released less than 10% of the original
oxygen available. Regardless of the length of operating time of
the Type II canisters, a constant CO_2 Absorbed/O_2 Released ratio of
0.44 was obtained from all canisters.

The data in Table 2 show clearly that when the user experienced
no undue strain in breathing (use time of 60 minutes) there was an
adequate quantity of oxygen released, about 2.4 liters/minute, and an
effective CO_2 absorption, about 1.1 liters/minute. However, in the
cases of the two subjects who were unable to continue breathing with
the OBA beyond 20 minutes the amounts of oxygen release and CO_2 absorbed
were markedly reduced; not including the oxygen released by the chlorate
candles the oxygen released was about 0.3 liters/minute and the CO_2
absorbed was about 0.1 liters/minute at a time-weighted-mean O_2 consump-
tion of 1.5 liters/minute work rate of man. Analyses of the chemical
reactions relative to the short operating canisters revealed the
chemical beds fully reacted with water in the expected manner. Hence,
it was implied that in these two cases the subjects were unable to force
their exhaled breaths into the KO_2 bed. As a result there was both
inadequate O_2 formation and CO_2 absorption. Naturally, under these
conditions the subjects were required to terminate the experiments.

DISCUSSION

Unused OBA Type II canisters have a relatively constant charge

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composition. This permits the analyses of used canisters for estimation of the residual oxygen and CO_2 absorbed due to manned-use to compare with the original amount of available oxygen and small quantity of CO_2 in unused canisters. These data permit one to determine whether the failure of a canister during use is due to depletion of the KO_2 charge, inadequacy of a given chemical reaction to go to completion when driven by man's respiration, or the inability of the user to force adequate volumes of expired air into the canister's chemical bed.

An increased breathing resistance, up to $+8.5$ dynes·sec·cm^{-5} during exhalation and down to -10.4 dynes·sec·cm^{-5} during inhalation, was the major cause for a user to change canisters in the OBA's employed in this study;[3] these breathing resistances represented greater than 21 to 270 percent changes, respectively, than permissible breathing resistance limits for closed-circuit breathing devices of this type.[3,4] As the airways of the OBA's employed were found to be unobstructed and all connections were free of leaks, the source of extreme breathing resistance was suspected to be within the specific Type II canisters. The excessive breathing resistances had to be due to either a failure of the chemical bed to absorb CO_2 or in an actual physical impairment of gas flow through the KO_2 bed; in either situation there would be a marked reduction in oxygen production and a CO_2 build-up within the facemask comparable with that actually observed.

Physical examination of the changes which occur with large

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KO_2 granules revealed that diffusion of exhaled water vapor into the centers of partially spent, large KO_2 granules did result in a "foaming" of the granule surface. Upon addition of more water the liberation of oxygen increased; however, in the manned-use situation the "foaming" could have significantly reduced pathways for gas flow and produced a high breathing resistance, which resulted in compounding the problem as less respiratory water could get to the KO_2 granules. Since either the build-up of CO_2 in the facemask or a great increase in breathing resistance, traceable to "foaming" of granules within the canister, would prevent continued use of the breathing apparatus, the OBA Model A-4 facemask was modified by using a close fitting, bivalved nosecup (Mine Safety Appliance "Nosecup" Part #457135) which permits both oral and nasal breathing. Several experiments were conducted equivalent to those when respiratory resistance forced premature termination of previous experiments. Results of the special manned OBA A-4 trials revealed outstanding performance of the total life support system while men performed heavy physical work in a hot-humid environment, Table 3. Apparently, the nosecup permits a more direct passage of respiratory water and CO_2, reducing the dead space within the mask and maintaining the more natural breathing pressures, from man to the chemical bed in the canister. Without the knowledge obtained from the chemical analyses of the canister charges and observation of "foaming" when insufficient water was available, in the presence of high CO_2, it would have been virtually impossible to have found a simple, inexpensive modification of the OBA to minimize the potential of what has been

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widely called "canister failure."

The conventionally used procedure for analysis of OBA canister contents[5] involves 1) removal of the KO_2 charge from the canister, 2) mixing the charge and trying to obtain a representative sample for analysis, 3) liberation of oxygen by water with the amount of oxygen measured with a flowmeter, 4) release of CO_2 by acidification of the solution from which oxygen was released, and 5) the measurement of evolved CO_2 volume with a flowmeter. The new method presented herein has many advantages in contrast with the conventional procedure. First, the new method does not require handling or sampling the very corrosive solid KO_2-KOH-K_2CO_3 mixture. Secondly, the entire contents of the canister are analyzed so variations within granules cannot lead to inaccurate samples. Absorption of both water and CO_2 from the atmosphere during analysis is virtually eliminated. Finally, the new method yields a much better estimate of the original available oxygen in the canister than may be derived from the weight of the used canister charge.

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LEGEND FOR FIGURE

Figure 1. Apparatus for analysis of Oxygen Breathing Apparatus

(OBA) canisters. The tank, T, contains a known volume of water

(30 liters) which serves to seal the bottom of the bell, B, and

to react with the canister charge. Holes punched in the canister,

C, allow rapid release of oxygen when the canister is submerged

within the bell. A 300 liter spirometer is used to collect and

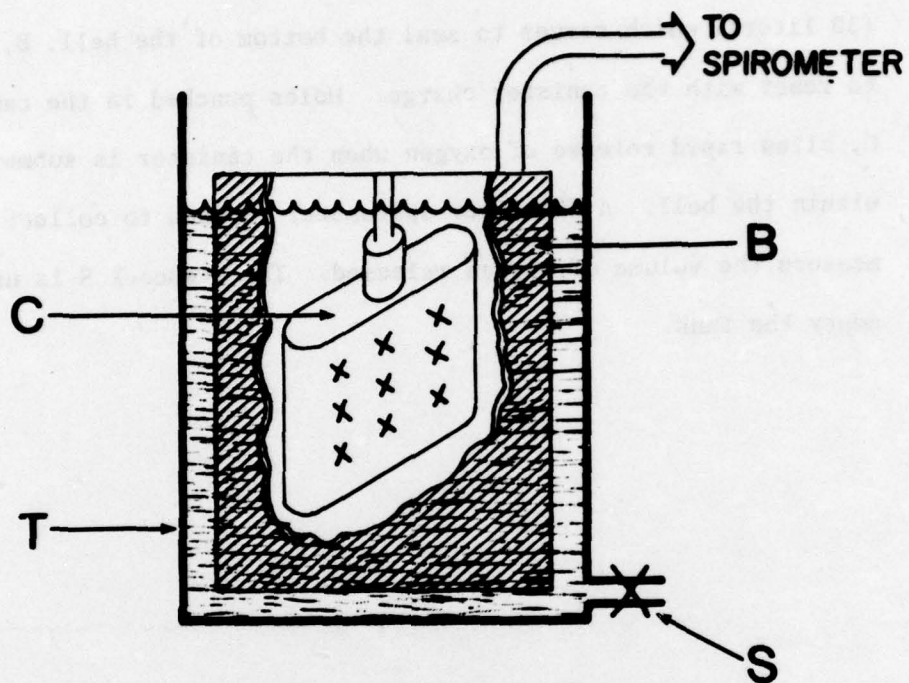measure the volume of oxygen released. The stopcock S is used to

empty the tank.

Figure 1

TABLE 1

ANALYSIS OF UNUSED OXYGEN BREATHING APPARATUS (OBA) CANISTERS

Manufacture Date	M_{CO_2} Moles	M_{KOH} Moles	Charge Weight Grams	Oxygen Released Moles	Theor. as KO_2* Moles	Apparent Purity as KO_2 %
12-1-73	0.09	13.39	915	9.21	10.04	91.7
11-15-73	0.08	13.02	899	9.05	9.77	92.7
3-13-74	0.12	13.83	987	9.45	10.37	91.1
3-13-74	0.05	13.31	914	9.14	9.98	91.6
3-13-74	0.06	13.33	915	9.16	9.92	91.6
3-13-74	0.06	13.43	920	9.22	10.07	91.5
3-14-74	0.05	13.36	916	9.23	10.02	92.1
3-13-74	0.05	13.78	937	9.42	10.33	91.1
11-13-74	0.07	13.36	911	9.14	9.85	92.8
3-30-73	0.15	14.81	1025	10.04	11.11	90.4
3-23-73	0.14	14.83	1025	9.79	11.12	88.0
3-27-73	0.18	14.83	1034	10.04	11.12	90.3
3-29-73	0.18	14.22	999	9.64	10.66	90.4
3-26-73	0.20	14.87	1037	10.04	11.15	90.0
3-15-73	0.18	14.97	1035	10.02	11.23	90.8
3-26-73	0.25	15.47	1088	10.43	11.60	89.9
3-20-73	0.17	14.88	1047	9.81	11.16	87.9
Mean	0.122		977	9.58		90.8
SD	0.063		63	0.43		1.4
SEM	0.014		15	0.10		0.33
Liters (STPD)						
Mean	2.7			214.6		
SD	1.4			9.6		
SEM	0.31			2.2		

*Theoretical Oxygen as M_{KO_2} = M_{KOH} X 0.75

Blank CO_2 = 0.122 Moles/Canister

TABLE 2

ANALYSIS OF USED TYPE II CANISTERS

Use Time (min.)	M_{KOH}	M_{CO_2}	Residual O_2 M	Original * O_2 M	O_2 Liberated M	CO_2 ** Absorbed M
60	7.25	2.85	2.45	8.68	6.23	2.73
60	7.81	2.97	2.97	9.22	6.25	2.85
60	8.64	2.99	3.19	9.81	6.62	2.87
23	14.58	0.41	9.73	10.34	0.61	0.29
20	11.81	0.47	7.71	8.54	0.83	0.35

* Calculated by Equation 9 with $M_{CO_2 blank}$ = 0.12

** M_{CO_2} - $M_{CO_2 blank}$

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TABLE 3

SPECIAL MANNED TRIALS OF OBA A-4 WITH TYPE II CANISTERS *

Manned Operation Time (mins.)		60
Nose Cup in Facemask		Yes
Amount of O_2 Generated (Liters STPD)		143.1
Percent O_2 Generated of Total O_2 Available (%)		72.3
Amount of O_2 Generated per Minute (Liters/min. STPD)		2.4
Amount of CO_2 Absorbed: Total (Liters STPD)		62.1
Per Minute (Liters/min. STPD)		1.04
Mean O_2 in Mask (%)		81.1
Mean CO_2 in Mask (%)		1.5

Breathing Resistances (dynes·sec·cm^{-5}):

Mean Inhalation Resistance During Physical Work	-1.5
Percent Change of Maximum Permissible Limit (%)	-46.6 **
Mean Exhalation Resistance During Physical Work	+5.7
Percent Change of Maximum Permissible Limit (%)	-19.1 **
Mean Inhalation Resistance During Standing Rest	-0.6
Percent Change of Maximim Permissible Limit (%)	-78.6 **
Mean Exhalation Resistance During Standing Rest	+4.3
Percent Change of Maximum Permissible Limit (%)	-39.0 **
Estimated Cardiovascular Reserve at End of Trails (%)	58

Maximum Canister Surface Temperatures:	°C	°F
Bottom (opposite chlorate candle)	60	140
Middle (along centerline)	255	491
Upper Quarter (along centerline)	168	334
Top (left side of neck)	122	252

*Time-weighted-mean O_2 consumption = 1.5 liters/minute STPD; all chlorate candle primers and candles and KO_2 beds operated to indicated percentages; n = 2 for special trials only.

** Negative values are indicative of resistance below the maximum permissible limits, therefore, all resistances are well within the permissible limits.